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Accomplishments

Title: Enhanced Photophysical Processes in Controlled Electromagnetic Nanoenvironments

Contract/Grant #: FA9550-06-1-0021

Summary

The objectives of this grant “Enhanced Photophysical Processes in Controlled Nanoenvironments” were to investigate the role of electromagnetic nanoenvironments on fundamental photophysical processes in molecular species. By systematically designing tunable plasmonic nanostructures such as nanoshell dimers, small clusters, extended monolayer arrays of nanoparticles, and other hybridized structures, controlled electromagnetic environments can be created. The enhanced fields generated in these environments is then used to study photophysical processes such as surface enhanced Raman scattering (SERS), Fluorescence enhancement and surface enhanced infrared absorption (SEIRA) of molecules placed in these enhanced fields. All the objectives stated were successfully achieved. Below is a brief description of the particular achievements. A complete list of publications is attached at the end.

Complex plasmonic nanostructures developed:

Nanoshells: The silica core-gold shell nanoshell is the workhorse substrate developed in the Halas group. This is a multistep process that requires the reactants to be aged and have limited useful shelf life. A new technique for reducing the gold shell was developed that is suitable for non optimal reactants and is general for coating various dielectric cores and shapes. (Brinson et al. 2008)

Mesoscopic solid nanoparticles and nanoshells: To investigate the tunability range of nanoparticles and nanoshells and the field enhancement in the near to mid IR range, colloidal solid gold nanoparticles in the 430 nm size regime and nanoshells of all sizes and aspect ratios were fabricated. Their field enhancements for surface enhanced spectroscopy were compared to theoretical predictions from Mie theory. The mesoscopic nanoparticles tune into the near IR and

can be used for SERS, whereas the large nanoshells although have resonances in the mid IR, do not have the field enhancement for SEIRA. (Wang et al. 2008; Tam et al. 2007)

Dimers: The field enhancement in the junction between two nanoparticles or nanostructures is greatly enhanced as compared to the surface of an isolated nanoparticle. Plasmonic dimer nanostructures were fabricated by a number of different methods and their ensemble and single particle properties were investigated. Structures investigated include nanoshell heterodimers, nanoshell homodimers, solid nanosphere and nanoshell homo and heterodimers, and a solid nanoparticle embedded in the core of a nanoshell. The last two structures were studied using single particle dark field microspectroscopy and show interesting coherent phenomenon a Fano resonance that has only recently been observed in plasmonic nanostructures. The Fano resonance is extremely sensitive to the embedding environment and maybe used for sensitive surface plasmon sensing applications. (Wang et al. 2006; Lassiter et al 2008; Brown et al. 2009; Bardhan et al. 2009)

Arrays : Extending the dimer geometry with a single junction with an enhanced near field, we have generated arrays of nanoparticles and nanoshells with sub 10nm gaps. The extended arrays of nanoshells have plasmon resonances in the near to mid IR and have been demonstrated as effective substrates for both SERS and SEIRA. Using para mercaptoaniline (pMA) as a test analyte molecule, conservative SERS enhancement factors of 10^6 and SEIRA enhancement factors of 10^4 have been demonstrated. The nanoshell array substrates reported herein provide a new, multifunctional platform for chemical sensing applications by enhancing both RS and IRA spectroscopy. Integrating SERS and SEIRA on a single substrate will enable the identification of unknown molecules by combining both surface-enhanced vibrational spectroscopies, allowing

more detailed investigations of molecular structure, orientation, and conformation, as well as adsorbate–substrate and adsorbate – adsorbate interactions. (Wang et al. 2007)

Gaps: Electromigrated gaps in gold wires were investigated as single molecule SERS substrates. This is the first demonstration of simultaneous observation of electrical conductance co related with optical SERS measurements. (Ward et al. 2007; Ward et. al. 2008)

Nanoholes: Plasmonic properties of nanoholes in a gold film were investigated using Plasmon Hybridization and correlated with experimentally fabricated structures. In addition techniques to create an array of holes and fill the holes with a controlled number of nanoparticles to create nanoparticle dimers trimers and larger aggregates were developed. (Park et al. 2008; Mirin et al. 2008)

Nanoparticle-nanowire and Nanoparticle-film hybrid structures: Placing a nanoparticle near a thin gold film changes the plasmonic interactions between the particle and the film. The strength of the interaction depends strongly upon the thickness of the film. These interactions were investigated with films of varying thickness with nanoparticles of two different sizes and the results correlated with theoretical calculations based on Plasmon Hybridization. This system was found to be analogous to the Anderson impurity problem. (Le et al. 2007) Another system investigated is a Ag nanoparticle near a Ag nanowire. The nanoparticle may be used to couple light in and out of the nanowire. The polarization dependence of the input light to the light output was investigated both experimentally and using finite element methods (COMSOL Multiphysics package). (Knight et al. 2007)

Enhanced spectroscopy:

Near-field of nanoshells: The strength and extent of the near field surrounding plasmonic nanoparticles determines the useful sensing volume of the nanostructure. A technique to determine the extent of the field is demonstrated using nanoshells and verified by correlating with Mie Theory simulations. This also leads to the development of a parameter L_{SERS} that can be used to determine the useful sensing volume. (Lal et al. 2006)

Metal enhanced Fluorescence: The fluorescence of dye molecules is enhanced when placed near a metal surface. The mechanism of enhancement and the contribution of the enhanced near field of the metal nanostructure in fluorescence enhancement is investigated by placing a weak dye Indocyanin Green (ICG) in the near field of a nanoshell. By varying the position of the nanoshell plasmon resonance and thus the near field experienced by the dye molecule the effect on the absorption and emission on the fluorescence enhancement has been investigated. We find that enhancement of the molecular fluorescence by more than a factor of 50 can be achieved for ICG next to a nanoparticle with a large scattering cross section and a plasmon resonance frequency corresponding to the emission frequency of the molecule. The distance dependence of the fluorescence enhancement as the dye molecule is placed at varying distances from the nanoshell surface has also been investigated by building thin silica epilayers on the nanoshell surface of varying thickness and placing the dye molecules on the silica epilayer. A maximum enhancement of $\sim 50\times$ is observed for silica layers of 7 nm thickness for weak dye molecules. (Tam et al. 2007, Bardhan et al. 2008)

In another series of experiments to examine the fluorescence enhancement of IR800 conjugated to nanoshells and nanorods, both NSs and NRs lead to large increases in quantum yield relative to the isolated fluorophore. We have observed that NSs are more efficient in improving the

emissive properties of a fluorophore due to their significant scattering cross section at the emission wavelength of the fluorophore. Additionally, the near-field response of NSs gives rise to a considerable enhancement in the absorption and the radiative decay rate of IR800, resulting in 40-fold enhancement and 86% quantum yield. IR800 molecules bound to NRs demonstrate a 9-fold emission enhancement and a 74% quantum yield, attributable to the high local field enhancement at the longitudinal plasmon wavelength. Therefore utilizing Au nanoparticles with appropriate geometry and dimensions for emission enhancement is a useful strategy for enhancing the detection sensitivity of low-quantum-yield fluorescent emitters. This approach is also potentially valuable in biomedical imaging and, moreover, can be conveniently generalized to enhance other fluorescent media. (Bardhan et al. 2009)

Raman Optical Activity: Measurements of near-infrared scattered circular polarization Raman optical activity (SCP-ROA) were made using laser excitation at 780 nm for samples of S-(-)- α -pinene and L-alanyl-L-alanine. These are the first measurements of ROA outside the blue-to-green visible region between 488 and 532 nm. Comparison of Raman and ROA intensities measured with excitation at 532 and 780 nm demonstrate that the expected frequency to the fourth-power dependence for Raman scattering and the corresponding fifth-power dependence for ROA are observed. These measurements were performed on the first NIR excitation ROA instrument developed in the Halas laboratory. (Nafie et al. 2007)

Surface enhanced Raman scattering (SERS): SERS is one of the best understood surface enhanced spectroscopy. It has been utilized effectively for small molecules. In the current set of investigations, the SERS of large biologically relevant molecules have been studied with nanoshells as substrates on which the molecules have been immobilized. SERS of DNA, peptides, aptamers, and other molecules that cannot be covalently linked to the gold surface such

as ibuprofen on nanoshells have been investigated. For large molecules that may adopt many conformations on the surface of a nanoshell the reproducibility of the SERS spectra is a big issue. A number of techniques to form better SAMS of the large molecule (DNA) were developed. In addition a cross correlation function was developed to quantify the reproducibility of the SERS spectra. This was then used to optically investigate the binding of aptamers with target molecules. (Wei et al. 2008, Barhoumi et al. 2008, Barhoumi et al. 2008, Neumann et al. 2009)

For molecules that do not bind to the nanoshell surface, a SAMS of a hybrid lipid bilayers was developed as a capture layer and the SERS spectra of the small molecules investigated. This technique is a general method that maybe used to immobilize small molecules within the near field of the nanoshell, and can be studied using SERS. (Levin et al. 2009)

Surface enhanced infra red absorption (SEIRA): Large nanoshells with resonances in the near to mid infrared were developed to investigate SEIRA using nanoshells as substrates. These proved to have very small SEIRA enhancements. As an alternative, aggregates of nanoshells with resonances in the near to mid IR were investigated as SEIRA substrates. These NS aggregates show SEIRA enhancements of $\sim 10^4$. SEIRA was then used to study intercalated ibuprofen in lipid bilayers, conformation of adenine and AMP molecules on nanoshells. (Kundu 2008, Levin 2009, Kundu 2009).

Applications:

Nanoshells can absorb light and effectively convert it to heat. This effect has been exploited in the photothermal therapy of cancer cells. Nanoshells have also been demonstrated as effective substrates for enhancing the fluorescence of weak dye molecules. Integrating multiple functionalities into individual nanoscale complexes is of tremendous importance in biomedicine,

expanding the capabilities of nanoscale structures to perform multiple parallel tasks. Here, the ability to enhance two different imaging technologies simultaneously—fluorescence optical imaging and magnetic resonance imaging—with antibody targeting and photothermal therapeutic actuation is combined all within the same nanoshell-based complex. The nanocomplexes are constructed by coating a gold nanoshell with a silica epilayer doped with Fe_3O_4 and the fluorophore ICG, which results in a high T_2 relaxivity ($390\text{mM}^{-1}\text{ s}^{-1}$) and 45 X fluorescence enhancement of ICG. Bioconjugate nanocomplexes target HER2+ cells and induce photothermal cell death upon near-IR illumination. (Bardhan 2009)

Another application of SERS has been in the study of the catalytic hydrodechlorination of 1,1-dichloroethene in H_2O . With Pd islands grown on Au nanoshell films, this reaction can be followed in situ using SERS, exploiting the high enhancements and large active area of Au nanoshell SERS substrates, the transparency of Raman spectroscopy to aqueous solvents, and the catalytic activity enhancement of Pd by the underlying Au metal. The formation and subsequent transformation of several adsorbate species was observed. These results provide the first direct evidence of the room-temperature catalytic hydrodechlorination of a chlorinated solvent, a potentially important pathway for groundwater cleanup, as a sequence of dechlorination and hydrogenation steps. More broadly, the results highlight the exciting prospects of studying catalytic processes in water in situ. (Heck et al. 2008)

Looking ahead we have studied the properties of nanoshell nanoparticle heterodimers and observed a plasmonic Fano resonance. This resonance is highly sensitive to the overlapping modes that give rise to such resonances. This could lead to sensitive dielectric environmental sensing modalities. (Brown et al. 2010)

Conclusion

The goals we had outlined in the original proposal have been well exceeded by the research and high impact publications that have originated due to this grant. Many new future directions of research have also emerged as a result of this grant. The three most interesting directions are the investigation of the coherent phenomenon and Fano resonances in asymmetric nanostructures and heterodimers which can lead to new sensitive sensing modalities. The next interesting direction is the exploiting the near field of nanoshells and other nanostructures to enhance fluorescence of weak FDA approved dye molecules for in vivo bioimaging and fluorescence optical tomography. The last direction is exploiting the enhanced near field of nanoshells to enhance the generation of excitons in quantum dots or other dye molecules for energy applications.

Complete List of Publications

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